1986

ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT

SECTION 7

DRINKING AND SURFACE WATER SAMPLES

INORGANIC TRACE CONTAMINANTS SECTION

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Ministry of the Environment

1986

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Laboratory Services Branch
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ALRP

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INORGANIC TRACE CONTAMINANTS SECTION

SUMMARY

I. Introduction

The Inorganic Trace Contaminants Section of the Ministry of the Environment, Laboratory Services Branch is responsible for the analysis of a wide variety of sample types for metals and non-metals. The use of sensitive instrumentation and methodologies appropriate to the sample matrix, combined with quality assurance programs, ensures that the Section is able to maintain a high standard of analytical performance. This performance is monitored through regular internal quality control and assurance programs as well as participation in interlaboratory round-robins. This QA report summarizes the methodologies used for analysis of these samples and the supporting internal quality assurance data.

This report is assembled in sections that reflect the analyses performed on different sample matrices in support of the programs of the Ministry of the Environment. Coincidentally, these divisions also reflect the supervisory responsibilities within the Section.

II. Quality Control and Assurance

The objectives of the quality control and assurance programs are to ensure that all of the components of the analytical process are under control and to ensure immediate detection and correction of unacceptable analytical performance. The program monitors all of the reagents, instrumentation, calibration and recovery components of the analytical system.

A. Quality Control

Quality control of the analytical process takes place at the instrument level and is intended to ensure that the instrumentation is operating according to established criteria. This control function ensures that instrument calibration, standardization, slope and intercept, and instrumental drift meet these criteria.

B. Quality Assurance

Quality assurance of the analytical process takes place after the results have been generated and is intended to ensure that the analytical protocols of sample preparation and digestion have been carried out correctly. This control function ensures that reagent blanks, digested standards, sample duplicates and recovery materials meet established response criteria.

III. Report Format

The report consists of one page method summaries and one page data summaries of blanks, between-run controls and within-run duplicates in formats that are common to all of the parameter/matrix combinations. The method summaries give a brief outline of the sample preparation and measurement procedures. The data summaries consist of annual mean values with standard deviations.

For the within-run duplicates, the data set is subdivided into ranges approximating 0 to 20 %, 20 to 50 % and 50 to 100% of the analytical range. All results for duplicates reported to the data base that are "<" or that have been diluted into the range are excluded from the statistical analysis.

The standard deviations for blanks and between-run controls are calculated using formula I. Formula II is used for the calculations for within-run duplicates.

$$sd = sqrt[\{(sumx2 - (sumx)2\}/n/(n-1)] \quad .....I$$

$$sd = sqrt(sumd2/2n) \quad .....II$$

where : x = the individual values; n = the number of events
d = the differences between pairs of duplicates

The data is stored in a personal computer using BMB Manager II files. All data manipulations, reports generated etc, are performed using applications written in Manager Math.

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7. Water Samples

7.1 Miscellaneous Water

Water samples for metals analyses, including drinking and surface water, are collected in plastic or glass containers and preserved with nitric acid. Samples for mercury are preserved with acidic potassium permanganate, while those for cyanide are preserved with sodium hydroxide (pH 12). Zinc acetate and sodium carbonate (>pH 10) are used to preserve samples for sulfide.

Table 7.1 presents the parameters determined, the type of sample digestion and the analytical instruments used in the analysis of water samples.

TABLE 7.1

Parameter	Collection Device	Preparation	Analysis
Metals	Glass or plastic bottles	Acid digest	AAS, ICP-AES
Mercury	Glass or plastic bottles	Acid digest	Cold Vapour AAS
Hydride Metals	Glass or plastic bottles	Acid digest	AAS
Free Cyanide	Glass or plastic bottles	Low T dist	Colorimetry
Total Cyanide	Glass or plastic bottles	Manual dist	Colorimetry
Boron	Plastic bottles	None	ICP-AES

7.2 Miscellaneous Water Quality Assurance

Sample duplicates are prepared by pouring a second aliquot from the sample bottle.

Reagent blanks are analysed with each analytical run. There are sufficient variations in the digestion acid lots that only one lot should be used in any one analytical run.

Matrix matched between-run composite samples are prepared by collecting samples in a large container. New composites are collected as the first is depleted or as the stability period expires. These composites may be spiked as necessary to provide a measureable level of analyte.

Table 7.2 indicates the sample descriptors used in the QA summary data, the source and the parameters that they are used to control.

TABLE 7.2

Sample Designation	Type	Parameter
qcs1	Multielement standard	Metals
QC	Fe and Mn standard	Fe, Mn
comp	Filtered composite spiked	Uranium
qca1,qcb1	0.1 ppm standard	Cyanide
qcd	0.2 ppm standard	Cyanide
con g2-3, g2-4	composite sample	Mercury
475-3	EPA standard solution	Arsenic
chk	Filtered composite spiked	Boron
epa1,epah	EPA standard solutions	Sb, Ag, Tl

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Aluminum TEST CODE: ALUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BE2
REVISION NO: Original DATE: April, 1985
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 1000 ml
Container- One litre plastic bottle with non-metallic cap liner
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pour sample into a 30 x 200 mm quartz test tube held in rack until lower miniscus of water column reaches 100 ml calibration mark. Add 2 ml 20% (v/v) HNO3 and evaporate to dryness in mechanical convection oven (with efficient exhaust to extract acidic vapours). Set effective temperature to 90 ± 5°C.
Prepare several runs and dry in oven. A typical LIS run consists of 38 tubes including 4 blanks, 3 QCs and 2 duplicate spikes (QC solution - 5 ml - added by Brinkmann dispenser). Cool, add 5 ml 5% HNO3 and 1 drop 50% H2O2. Seal tubes with Parafilm, stir with Vortex and hand-turn to wet entire inside surface. Repeat after ½ hour. Transfer supernatant to numbered tube (17 x 100 mm) with cap.
INTERFERENCES: Several; compensated for by computer program.

REPORTING RESULTS:2 sig. figs. if > 0.010 µg/ml;if < 0.010 -1 sig fig.
INSTRUMENTATION: Jarell Ash Atom-Comp -ICP Model 975

Calibration Range: 0.08 to 500 mg/L
Resolution: 0.01 mg/L
Sensitivity:
Instrument Detection Limit: 0.08 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 1.000 mg/L
Accuracy-104.1% (QCS, in-house control, X = 0.052 µg/ml, N = 36)
Precision of Controls-

	A	B
mean	1.057mg/L	
std. dev.	0.111mg/L	
R.S.D.	10.5 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.006	0.064	0.035
mean	0.100	0.302	0.673

W .01 mg/L T .10 mg/L

CONTROL LIMITS:

REMARKS: P-E 5000 AAS is available as back-up unit.
In the case of throughfalls, terrestrial effects and other APIOS samples, concentrate 50 ml down to 5 ml.

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINIUM IN MISCELLANEOUS WATERS

Operating Range = .00300to 1.000 mg/L

IN - RUN DUPLICATES

range	<.00300	.00300to 0.200	0.200to 0.500	0.500to 1.00	> 1.00
no.	4	221	72	41	13
s.w.		0.00640	0.06420	0.03470	
mean		0.10030	0.30240	0.67250	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcs1	1056	1.05690	0.11124	10.53

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	40	.00868	.00598

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:ARSENIC TEST CODE:ASUT SAMPLE TYPE: Water
UNIT: Biomaterials SUPERVISOR: R. Sadana

METHOD CODE:510CF3
REVISION NO: DATE: January 1983
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 100 ml
Container- Glass bottle with bakelite screw cap (16 oz.)
Preservative-1ml conc. HNO3 for sample filling 16 oz. bottle
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-yes % Extracted->90%
Procedure-A twenty ml sample is pipetted into 20x150 mm pyrex test tube. A batch of sixty-eight tubes including samples,blanks, standards and controls are run. These samples are fed to a automated system for measurement of arsenic by hydride-FAAS technique.
Samples with arsenic concentration exceeding 10 ng/ml are digested by pipetting 20 ml of sample in a 100 ml beaker and adding 4 ml 6:3:1 HNO3:HCLO4:H2SO4. Heat until dense white fumes evolve. Cool, add 0.5 ml of H2O and 2.5 ml of HCl. Transfer the digestate to a test tube calibrated at 20 ml, dilute to mark with DDW, mix well, and analyze.

INTERFERENCES: Excessive concentrations of Cu, Fe, Ni

REPORTING RESULTS:mg/L-2 dec. if <10, 1 dec. if 10-100, 0 dec. if >100
INSTRUMENTATION: Atomic Absorption Spectrophotometer (Varian 1200) with strip chart recorder, peristaltic pump, auto-sampler, open-ended and heated quartz 'T' cell (0.6 x 10 cm), and gas-liquid separator.
Calibration Range: 0 to 40 ng/ml
Resolution: 0.01 absorbance
Sensitivity: 20 ng/ml reads 0.150 abs.
Instrument Detection Limit: 1 ng/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.001 to 0.04 mg/L
Accuracy-
Precision of Controls-

	A	B
mean	.413 mg/L	
std. dev.	.011 mg/L	
R.S.D.	2.7 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	.0000	0.0004	0.0005
mean	.002	.011	0.025

W .001 mg/L T .005 mg/L

CONTROL LIMITS:

REMARKS:Accuracy = ratio of mean to cert. value of ref. std. X 100

SUMMARY REPORT OF QUALITY CONTROL DATA

ARSENIC IN MISCELLANEOUS WATERS

Operating Range = .00100to 0.040 mg/L

IN - RUN DUPLICATES

range	<.00100	.00100to 0.008	0.008to 0.020	0.020to 0.04	> 0.04
no.	89	18	3	2	0
s.w.		0.00000	0.00040	0.00050	
mean		0.00200	0.01100	0.02500	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
475-5	51	0.03500	0.00150	4.29

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	.00000	.00000

DATE 87/03/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Barium TEST CODE: BAUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BE2
REVISION NO: Original DATE: April, 1985
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 1000 ml
Container- One litre plastic bottle with non-metallic cap liner
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pour sample into a 30 x 200 mm quartz test tube held in rack until lower miniscus of water column reaches 100 ml calibration mark. Add 2 ml 20% (v/v) HNO3 and evaporate to dryness in mechanical convection oven (with efficient exhaust to extract acidic vapours). Set effective temperature to 90 ± 5°C.
Prepare several runs and dry in oven. A typical LIS run consists of 38 tubes including 4 blanks, 3 QCs and 2 duplicate spikes (QC solution - 5 ml - added by Brinkmann dispenser). Cool, add 5 ml 5% HNO3 and 1 drop 50% H2O2. Seal tubes with Parafilm, stir with Vortex and hand-turn to wet entire inside surface. Repeat after ½ hour. Transfer supernatant to numbered tube (17 x 100 mm) with cap.
INTERFERENCES: Several; compensated for by computer program.

REPORTING RESULTS:2 sig. figs. if > 0.010 µg/ml;if < 0.010 -1 sig fig.
INSTRUMENTATION: Jarell Ash Atom-Comp -ICP Model 975

Calibration Range: 0.008 to 75 mg/L
Resolution: 0.001 mg/L
Sensitivity:
Instrument Detection Limit: 0.008 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.100 mg/L
Accuracy-83.6% (QCS, in-house control, X = 0.0084 mg/L, N = 36)
Precision of Controls-

	A	B
mean	.221 mg/L	
std. dev.	.0187mg/L	
R.S.D.	8.5 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0012	0.0022	0.0058
mean	0.014	0.031	0.066

W .001 mg/L T .010 mg/L

CONTROL LIMITS:

REMARKS: P-E 5000 AAS is available as back-up unit.
In the case of throughfalls, terrestrial effects and other APIOS samples, concentrate 50 ml down to 5 ml.

SUMMARY REPORT OF QUALITY CONTROL DATA

BARIUM IN MISCELLANEOUS WATERS

Operating Range = .00050to 0.100 mg/L

IN - RUN DUPLICATES

range	<.00050	.00050to 0.020	0.020to 0.050	0.050to 0.10	> 0.10
no.	4	65	183	41	58
s.w.		0.00120	0.00220	0.00580	
mean		0.01410	0.03080	0.06640	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcs1	1061	0.22080	0.01866	8.45

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	33	.00148	.00174

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Beryllium TEST CODE: BEUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BE2
REVISION NO:2 DATE: January 15, 1986
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 1000 ml
Container- One litre plastic bottle with non-metallic cap liner.
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pour sample into a 30 x 200 mm quartz test tube held in rack until lower miniscus of water column reaches 100 ml calibration mark. Add 2 ml 20% (v/v) HNO3 and evaporate to dryness in mechanical convection oven (with efficient exhaust to extract acidic vapours). Set effective temperature to 90 ± 5°C.
Prepare several runs and dry in oven. A typical LIS run consists of 38 tubes including 4 blanks, 3 QCs and 2 duplicate spikes (QC solution - 5 ml - added by Brinkmann dispenser). Cool, add 5 ml 5% HNO3 and 1 drop 50% H2O2. Seal tubes with Parafilm, stir with Vortex and hand-turn to wet entire inside surface. Repeat after ½ hour. Transfer supernatant to numbered tube (17 x 100 mm) with cap.
INTERFERENCES: Several (compensated for by computer program).

REPORTING RESULTS:Report > 0.010 ug/ml to 2 sig figs;< 0.010 to 1 sig.
INSTRUMENTATION: Jarrell Ash Atom-Comp ICP Model 975.

Calibration Range: 0 to 75 mg/L
Resolution: 0.001 mg/L
Sensitivity:
Instrument Detection Limit: 0.009 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.100 mg/L
Accuracy- 91.5% (QCS, in-house control; X = 0.18 mg/L; N = 109)
Precision of Controls- A B

mean	.2174mg/L
std. dev.	.0150mg/L
R.S.D.	6.9 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0009	0.0004	
mean	0.0104	0.0220	

W .0005mg/L T .0050mg/L

CONTROL LIMITS:

REMARKS:PE - 5000 AAS available as back-up unit.
For throughfalls, terrestial effect and other APIOS samples, 50 ml volume is concentrated to 5 ml.

SUMMARY REPORT OF QUALITY CONTROL DATA

BERYLLIUM IN MISCELLANEOUS WATERS

Operating Range = .00050to 0.100 mg/L

IN - RUN DUPLICATES

range	<.00050	.00050to 0.020	0.020to 0.050	0.050to 0.10	> 0.10
no.	75	266	10	0	0
s.w.		0.00090	0.00040	0.00000	
mean		0.01040	0.02200	0.00000	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcs1	865	0.21740	0.01500	6.90

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	.00000	.00000

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Boron TEST CODE: BBUT SAMPLE TYPE: Water
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:001AE2
REVISION NO: 1 DATE: February 1, 1987
NATURE OF LAST REVISION:The ICP-AES replaced azomethine-H colorometric

SAMPLE HANDLING:
Quantity Required- 500 ml
Container- Polyethylene container
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted->100
Procedure-The samples are analyzed straight without pretreatment. No processing is required.

INTERFERENCES:None
waste matrices.
REPORTING RESULTS: mg/l
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400, equipped with autosampler and DEC computer system for concentration print-out, Apple microcomputer interface to LIS.
Calibration Range: 0 to 1.000 mg/L
Resolution:
Sensitivity: 1.00 mg/L standard gives 3000 counts.
Instrument Detection Limit: 0.05 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.001 to 1.00 mg/L
Accuracy-
Precision of Controls-

	A	B
mean	.109 mg/L	
std. dev.	.016 mg/L	
R.S.D.	15.1 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.010		0.009
mean	0.038		0.536

W .005 mg/L T .050 mg/L

CONTROL LIMITS:

REMARKS: Replaces Azomethine-H colorometric method.

SUMMARY REPORT OF QUALITY CONTROL DATA

BORON-JA IN MISCELLANEOUS WATERS

Operating Range = .00100to 0.100 mg/L

IN - RUN DUPLICATES

range	<.00100	.00100to 0.020	0.020to 0.050	0.050to 0.10	> 0.10
no.	34	218	48	28	23
s.w.		0.00220	0.01670	0.02160	
mean		0.00660	0.03160	0.07140	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	226	.00147	.00197

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Calcium TEST CODE: CAUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BE2
REVISION NO:2 DATE: January 15, 1986
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 1000 ml
Container- One litre plastic bottle with non-metallic cap liner.
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pour sample into a 30 x 200 mm quartz test tube held in rack until lower miniscus of water column reaches 100 ml calibration mark. Add 2 ml 20% (v/v) HNO3 and evaporate to dryness in mechanical convection oven (with efficient exhaust to extract acidic vapours). Set effective temperature to 90 ± 5°C.
Prepare several runs and dry in oven. A typical LIS run consists of 38 tubes including 4 blanks, 3 QCs and 2 duplicate spikes (QC solution - 5 ml - added by Brinkmann dispenser). Cool, add 5 ml 5% HNO3 and 1 drop 50% H2O2. Seal tubes with Parafilm, stir with Vortex and hand-turn to wet entire inside surface. Repeat after ½ hour. Transfer supernatant to numbered tube (17 x 100 mm) with cap.
INTERFERENCES: Several (compensated for by computer program).

REPORTING RESULTS: Whole numbers
INSTRUMENTATION: Jarrell Ash Atom-Comp ICP Model 975.

Calibration Range: 0 to 50 mg/L
Resolution: 0.02 mg/L
Sensitivity:
Instrument Detection Limit: 0.02 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 50.0 mg/L
Accuracy- 92.0% (QCS, in-house control; X = 1.15 mg/l; N = 36)
Precision of Controls-

	A	B
mean	26.2 mg/L	
std. dev.	2.61mg/L	
R.S.D.	10.0 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.30	0.41	0.82
mean	4.1	16.6	36.1

W .2 mg/L T 1.0 mg/L

CONTROL LIMITS:

REMARKS: This test is performed in Water Quality Section and results reported as routine parameter.

SUMMARY REPORT OF QUALITY CONTROL DATA

CALCIUM IN MISCELLANEOUS WATERS

Operating Range = .00100to 50.000 mg/L

IN - RUN DUPLICATES

range	<.00100	.00100to10.000	10.000to25.000	25.000to 50.00	> 50.00
no.	1	80	55	86	129
s.w.		0.29420	0.41190	0.82240	
mean		3.98990	16.59550	36.05340	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcs1	1062	26.17200	2.61160	9.98

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	290	.00795	.02942

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Cadmium TEST CODE: CDUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BE2
REVISION NO:2 DATE: January 15, 1986
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 1000 ml
Container- One litre plastic bottle with non-metallic cap liner.
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pour sample into a 30 x 200 mm quartz test tube held in rack until lower miniscus of water column reaches 100 ml calibration mark. Add 2 ml 20% (v/v) HNO3 and evaporate to dryness in mechanical convection oven (with efficient exhaust to extract acidic vapours). Set effective temperature to 90 ± 5°C.
Prepare several runs and dry in oven. A typical LIS run consists of 38 tubes including 4 blanks, 3 QCs and 2 duplicate spikes (QC solution - 5 ml - added by Brinkmann dispenser). Cool, add 5 ml 5% HNO3 and 1 drop 50% H2O2. Seal tubes with Parafilm, stir with Vortex and hand-turn to wet entire inside surface. Repeat after ½ hour. Transfer supernatant to numbered tube (17 x 100 mm) with cap.
INTERFERENCES: Several (compensated for by computer program).

REPORTING RESULTS:Report > 0.010 ug/ml to 2 sig figs;< 0.010 to 1 sig.
INSTRUMENTATION: Jarrell Ash Atom-Comp ICP Model 975.

Calibration Range: 0.005 to 75 mg/L
Resolution: 0.001 mg/L
Sensitivity:
Instrument Detection Limit: 0.005 mg/L
PERFORMANCE CHARACTERISTICS:
Routire Operating Range- 0.0 to 0.020 mg/L
Accuracy- 94.4% (QCS, in-house control; X 0.0094 mg/L; N = 35)
Precision of Controls- A B
mean .1870mg/L
std. dev. .0124mg/L
R.S.D. 6.6 %

Precision of Duplicates-	low range	mid range	high range
s.d.	0.0016	0.0007	0.0014
mean	0.0011	0.0086	0.0134

W .0002mg/L T .0010mg/L

CONTROL LIMITS:

REMARKS:PE - 5000 AAS available as back-up unit.
For throughfalls, terrestial effect and other APIOS samples, 50 ml volume is concentrated to 5 ml.

SUMMARY REPORT OF QUALITY CONTROL DATA

CADMIUM IN MISCELLANEOUS WATERS

Operating Range = .00020to 0.020 mg/L

IN - RUN DUPLICATES

range	<.00020	.00020to 0.004	0.004to 0.010	0.010to 0.02	> 0.0
no.	14	10	248	34	45
s.w.		0.00160	0.00070	0.00140	
mean		0.00110	0.00860	0.01340	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcs1	1064	0.18700	0.01240	6.63

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	9	.00184	.00171

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Chromium TEST CODE: CRUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BE2
REVISION NO:2 DATE: January 15, 1986
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 1000 ml
Container- One litre plastic bottle with non-metallic cap liner.
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pour sample into a 30 x 200 mm quartz test tube held in rack until lower miniscus of water column reaches 100 ml calibration mark. Add 2 ml 20% (v/v) HNO3 and evaporate to dryness in mechanical convection oven (with efficient exhaust to extract acidic vapours). Set effective temperature to 90 ± 5°C.
Prepare several runs and dry in oven. A typical LIS run consists of 38 tubes including 4 blanks, 3 QCs and 2 duplicate spikes (QC solution - 5 ml - added by Brinkmann dispenser). Cool, add 5 ml 5% HNO3 and 1 drop 50% H2O2. Seal tubes with Parafilm, stir with Vortex and hand-turn to wet entire inside surface. Repeat after ½ hour. Transfer supernatant to numbered tube (17 x 100 mm) with cap.
INTERFERENCES: Several (compensated for by computer program).

REPORTING RESULTS:Report > 0.010 ug/ml to 2 sig figs;< 0.010 to 1 sig.
INSTRUMENTATION: Jarrell Ash Atom-Comp ICP Model 975.

Calibration Range: 0.01 to 75 mg/L
Resolution: 0.001 mg/L
Sensitivity:
Instrument Detection Limit: 0.01 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.100 mg/L
Accuracy- 96.7% (QCS, in-house control; X 0.012 mg/L; N = 36)
Precision of Controls-

	A	B
mean	.244 mg/L	
std. dev.	.0237mg/L	
R.S.D.	9.7 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0011	0.0012	0.0049
mean	0.012	0.027	0.093

W .0005mg/L T .0025mg/L

CONTROL LIMITS:

REMARKS:PE - 5000 AAS available as back-up unit.
For throughfalls, terrestial effect and other APIOS samples, 50 ml volume is concentrated to 5 ml.

SUMMARY REPORT OF QUALITY CONTROL DATA

CHROMIUM IN MISCELLANEOUS WATERS

Operating Range = .00050to 0.100 mg/L

IN - RUN DUPLICATES

range	<.00050	.00050to 0.020	0.020to 0.050	0.050to 0.10	> 0.10
no.	15	272	20	1	43
s.w.		0.00110	0.00120	0.00490	
mean		0.01150	0.02650	0.09250	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcs1	1062	0.24390	0.02368	9.71

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	33	.00148	.00177

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Cobalt TEST CODE: COUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BE2
REVISION NO: Original DATE: April, 1985
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 1000 ml
Container- One litre plastic bottle with non-metallic cap liner
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pour sample into a 30 x 200 mm quartz test tube held in rack until lower miniscus of water column reaches 100 ml calibration mark. Add 2 ml 20% (v/v) HNO3 and evaporate to dryness in mechanical convection oven (with efficient exhaust to extract acidic vapours). Set effective temperature to 90 ± 5°C.
Prepare several runs and dry in oven. A typical LIS run consists of 38 tubes including 4 blanks, 3 QCs and 2 duplicate spikes (QC solution - 5 ml - added by Brinkmann dispenser). Cool, add 5 ml 5% HNO3 and 1 drop 50% H2O2. Seal tubes with Parafilm, stir with Vortex and hand-turn to wet entire inside surface. Repeat after ½ hour. Transfer supernatant to numbered tube (17 x 100 mm) with cap.
INTERFERENCES: Several; compensated for by computer program.

REPORTING RESULTS:2 sig. figs. if > 0.010 µg/ml;if < 0.010 -1 sig fig.
INSTRUMENTATION: Jarell Ash Atom-Comp -ICP Model 975

Calibration Range: 0.02 to 75 mg/L
Resolution: 0.001 mg/L
Sensitivity:
Instrument Detection Limit: 0.02 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.100 mg/L
Accuracy- 95.7% (QCS, in-house control, X = 0.012 µg/ml, N = 35)
Precision of Controls-

	A	B
mean	.2527mg/L	
std. dev.	.0195mg/L	
R.S.D.	7.7 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0012	0.0011	0.0132
mean	0.0115	0.0245	0.0990

W .0005mg/L T .0025mg/L

CONTROL LIMITS:

REMARKS: P-E 5000 AAS is available as back-up unit.
In the case of throughfalls, terrestrial effects and other APIOS samples, concentrate 50 ml down to 5 ml.

SUMMARY REPORT OF QUALITY CONTROL DATA

COBALT IN MISCELLANEOUS WATERS

Operating Range = .00050to 0.100 mg/L

IN - RUN DUPLICATES

range	<.00050	.00050to 0.020	0.020to 0.050	0.050to 0.10	> 0.10
no.	21	273	14	1	42
s.w.		0.00120	0.00110	0.01320	
mean		0.01150	0.02450	0.09900	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcs1	1060	0.25270	0.01952	7.72

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	7	.00144	.00045

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Copper TEST CODE: CUUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BE2
REVISION NO:2 DATE: January 15, 1986
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 1000 ml
Container- One litre plastic bottle with non-metallic cap liner.
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pour sample into a 30 x 200 mm quartz test tube held in rack until lower miniscus of water column reaches 100 ml calibration mark. Add 2 ml 20% (v/v) HNO3 and evaporate to dryness in mechanical convection oven (with efficient exhaust to extract acidic vapours). Set effective temperature to 90 ± 5°C.
Prepare several runs and dry in oven. A typical LIS run consists of 38 tubes including 4 blanks, 3 QCs and 2 duplicate spikes (QC solution - 5 ml - added by Brinkmann dispenser). Cool, add 5 ml 5% HNO3 and 1 drop 50% H2O2. Seal tubes with Parafilm, stir with Vortex and hand-turn to wet entire inside surface. Repeat after ½ hour. Transfer supernatant to numbered tube (17 x 100 mm) with cap.
INTERFERENCES: Several (compensated for by computer program).

REPORTING RESULTS:Report > 0.010 ug/ml to 2 sig figs;< 0.010 to 1 sig.
INSTRUMENTATION: Jarrell Ash Atom-Comp ICP Model 975.

Calibration Range: 0.006 to 75 mg/L
Resolution: 0.001 mg/L
Sensitivity:
Instrument Detection Limit: 0.006 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.100 mg/L
Accuracy- 98.6% (QCS, in-house control; X 0.0099 mg/L; N = 34)
Precision of Controls-

	A	B
mean	.194 mg/L	
std. dev.	.0188mg/L	
R.S.D.	9.7 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0009	0.0019	0.0031
mean	0.011	0.029	0.084

W .0005mg/L T .0025mg/L

CONTROL LIMITS:

REMARKS:PE - 5000 AAS available as back-up unit.
For throughfalls, terrestial effect and other APIOS samples, 50 ml volume is concentrated to 5 ml.

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER IN MISCELLANEOUS WATERS

Operating Range = .00030to 0.100 mg/L

IN - RUN DUPLICATES

range	<.00030	.00030to 0.020	0.020to 0.050	0.050to 0.10	> 0.10
no.	3	236	44	30	38
s.w.		0.00090	0.00190	0.00310	
mean		0.01080	0.02920	0.08350	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcs1	1065	0.19390	0.01884	9.72

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	192	.00061	.00044

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Iron
UNIT: Water
TEST CODE: FEUT SAMPLE TYPE: Waters
SUPERVISOR: P. Vijan

METHOD CODE:522BA0
REVISION NO: 85-1
NATURE OF LAST REVISION:
DATE: July, 1985

SAMPLE HANDLING:
Quantity Required- 500 ml
Container- Plastic bottle with non-metallic cap liner
Preservative- 0.5 ml conc HNO3
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pipette 25 ml sample into a 1 oz flint glass vial with polyethylene lined screw cap. Add 0.5 ml 20% HNO3 to vial. Prepare several sets of 36 vials including 2 blanks, 2 in-house QC and 2 within-run duplicates for LIS runs. Dry overnight at 85 ± 5°C in forced air convection oven. Cool to room temperature and add 2.5 ml 5% (V/V) HCl to each vial by Oxford dispenser. Shake thoroughly to dissolve soluble salts. Determine Fe and Mn by AAS using composite standards (0.4, 1.0, & 2 ppm Mn; 2.0, 5.0 & 10.0 ppm Fe). The computer program allows direct input of results into LIS if P-E 5000 automated AAS system is used.
INTERFERENCES: Ca and Mg can cause severe interference if flame height and air-acetylene ratio are not optimized,
REPORTING RESULTS:2 dec. places if > 1 µg/ml;if < 1 - 3 dec. places
INSTRUMENTATION: Perkin-Elmer P-E 5000 AAS interfaced with PET computer.

Calibration Range: 0 to 10 µg/ml
Resolution:
Sensitivity: 0.12 µg/ml
Instrument Detection Limit: 0.05 µg/ml

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 1.000 mg/L
Accuracy- 101% (in-house standard, X = 0.202 mg/L, N = 67)
Precision of Controls-

	A	B
mean	.204 mg/L	
std. dev.	.0181mg/L	
R.S.D.	8.9 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.033	0.070	0.056
mean	0.065	0.316	0.708

W .02 mg/L T .10 mg/L

CONTROL LIMITS:

REMARKS: ICP-AES system may also be used for simultaneous measurement of both Fe and Mn with comparable accuracy and precision.

SUMMARY REPORT OF QUALITY CONTROL DATA

AA-IRON IN MISCELLANEOUS WATERS

Operating Range = .00500to 1.000 mg/L

IN - RUN DUPLICATES

range	<.00500	.00500to 0.200	0.200to 0.500	0.500to 1.00	> 1.00
no.	7	106	69	24	19
s.w.		0.03340	0.06980	0.05590	
mean		0.06480	0.31610	0.70790	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QC	437	0.20420	0.01810	8.86

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	441	.14280	.15970

DATE 86/09/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Iron TEST CODE: FEUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BA0
REVISION NO:2 DATE: January 15, 1986
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 1000 ml
Container- One litre plastic bottle with non-metallic cap liner.
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pour sample into a 30 x 200 mm quartz test tube held in rack until lower miniscus of water column reaches 100 ml calibration mark. Add 2 ml 20% (v/v) HNO3 and evaporate to dryness in mechanical convection oven (with efficient exhaust to extract acidic vapours). Set effective temperature to 90 ± 5°C.
Prepare several runs and dry in oven. A typical LIS run consists of 38 tubes including 4 blanks, 3 QCs and 2 duplicate spikes (QC solution - 5 ml - added by Brinkmann dispenser). Cool, add 5 ml 5% HNO3 and 1 drop 50% H2O2. Seal tubes with Parafilm, stir with Vortex and hand-turn to wet entire inside surface. Repeat after ½ hour. Transfer supernatant to numbered tube (17 x 100 mm) with cap.
INTERFERENCES: Several (compensated for by computer program).

REPORTING RESULTS:Report > 0.010 ug/ml to 2 sig figs;< 0.010 to 1 sig.
INSTRUMENTATION: Jarrell Ash Atom-Comp ICP Model 975.

Calibration Range: 0 to 50 mg/L
Resolution: 0.001 mg/L
Sensitivity:
Instrument Detection Limit: 0.02 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 1.000 mg/L
Accuracy- 92.9% (QCS, in-house control; X 0.046 mg/L; N = 36)
Precision of Controls-

	A	B
mean	1.00 mg/L	
std. dev.	0.134mg/L	
R.S.D.	13.4 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0099	0.0560	0.0370
mean	0.092	0.318	0.687

W .02 mg/L T .10 mg/L

CONTROL LIMITS:

REMARKS:
For throughfalls, terrestial effect and other APIOS samples, 50 ml volume is concentrated to 10 ml.

SUMMARY REPORT OF QUALITY CONTROL DATA

IRON IN MISCELLANEOUS WATERS

Operating Range = .00100to 1.000 mg/L

IN - RUN DUPLICATES

range	<.00100	.00100to 0.200	0.200to 0.500	0.500to 1.00	> 1.0
no.	1	176	91	51	32
s.w.		0.00990	0.05600	0.03700	
mean		0.09210	0.31840	0.68650	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcs1	1000	1.00020	0.13426	13.42

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	379	.02106	.08022

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Lead TEST CODE: PBUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BE2
REVISION NO:2 DATE: January 15, 1986
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 1000 ml
Container- One litre plastic bottle with non-metallic cap liner.
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pour sample into a 30 x 200 mm quartz test tube held in rack until lower miniscus of water column reaches 100 ml calibration mark. Add 2 ml 20% (v/v) HNO3 and evaporate to dryness in mechanical convection oven (with efficient exhaust to extract acidic vapours). Set effective temperature to 90 ± 5°C.
Prepare several runs and dry in oven. A typical LIS run consists of 38 tubes including 4 blanks, 3 QCs and 2 duplicate spikes (QC solution - 5 ml - added by Brinkmann dispenser). Cool, add 5 ml 5% HNO3 and 1 drop 50% H2O2. Seal tubes with Parafilm, stir with Vortex and hand-turn to wet entire inside surface. Repeat after ½ hour. Transfer supernatant to numbered tube (17 x 100 mm) with cap.
INTERFERENCES: Several (compensated for by computer program).

REPORTING RESULTS:Report > 0.010 ug/ml to 2 sig figs;< 0.010 to 1 sig.
INSTRUMENTATION: Jarrell Ash Atom-Comp ICP Model 975.

Calibration Range: 0 to 100 mg/L
Resolution: 0.001 mg/L
Sensitivity:
Instrument Detection Limit: 0.02 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.100 mg/L
Accuracy- 106% at 0.46 µg/ml
Precision of Controls-

	A	B
mean	.266 mg/L	
std. dev.	.054 mg/L	
R.S.D.	20.2 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.003	0.008	0.015
mean	0.012	0.030	0.082

W .005 mg/L T .025 mg/L

CONTROL LIMITS:

REMARKS:PE - 5000 AAS available as back-up unit.
For throughfalls, terrestial effect and other APIOS samples, 50 ml volume is concentrated to 5 ml.

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD IN MISCELLANEOUS WATERS

Operating Range = .00100to 0.100 mg/L

IN - RUN DUPLICATES

range	<.00100	.00100to 0.020	0.020to 0.050	0.050to 0.10	> 0.10
no.	39	235	28	14	35
s.w.		0.00300	0.00820	0.01520	
mean		0.01230	0.03030	0.08220	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcs1	1061	0.26600	0.05369	20.18

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	23	.00506	.00390

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Magnesium TEST CODE: MGUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BE2
REVISION NO: Original DATE: March, 1985
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 1000 ml
Container- One litre plastic bottle with non-metallic cap liner
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pour sample into a 30 x 200 mm quartz test tube held in rack until lower miniscus of water column reaches 100 ml calibration mark. Add 2 ml 20% (v/v) HNO3 and evaporate to dryness in mechanical convection oven (with efficient exhaust to extract acidic vapours). Set effective temperature to 90 ± 5°C.
Prepare several runs and dry in oven. A typical LIS run consists of 38 tubes including 4 blanks, 3 QCs and 2 duplicate spikes (QC solution - 5 ml - added by Brinkmann dispenser). Cool, add 5 ml 5% HNO3 and 1 drop 50% H2O2. Seal tubes with Parafilm, stir with Vortex and hand-turn to wet entire inside surface. Repeat after ½ hour. Transfer supernatant to numbered tube (17 x 100 mm) with cap.
INTERFERENCES: Several; compensated for by computer program.

REPORTING RESULTS: Whole numbers
INSTRUMENTATION: Jarell Ash Atom-Comp -ICP Model 975

Calibration Range: 0 to 20 mg/L
Resolution: 0.01 mg/L
Sensitivity:
Instrument Detection Limit: 0.005 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 50.000 mg/L
Accuracy- 95.9% (QCS, in-house control, X = 0.48 mg/L, N = 36)
Precision of Controls-

	A	B
mean	11.2 mg/L	
std. dev.	0.90 mg/L	
R.S.D.	8.0 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.11	1.84	1.07
mean	4.47	14.6	33.1

W .05 mg/L T .50 mg/L

CONTROL LIMITS:

REMARKS: This test is performed in the Water Quality Section and results reported as routine parameter.

SUMMARY REPORT OF QUALITY CONTROL DATA

MAGNESIUM-2 IN MISCELLANEOUS WATERS

Operating Range = .50000to 50.000 mg/L

IN - RUN DUPLICATES

range	<.50000	.50000to10.000	10.000to25.000	25.000to 50.00	> 50.00
no.	10	205	88	43	5
s.w.		0.16960	1.83440	1.37250	
mean		4.63230	17.04460	32.83760	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcs1	1062	10.43200	0.78570	7.53

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	600	.00770	.00684

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Manganese TEST CODE: MNUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BA0
REVISION NO:2 DATE: January 15, 1986
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 1000 ml
Container- One litre plastic bottle with non-metallic cap liner.
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pour sample into a 30 x 200 mm quartz test tube held in rack until lower miniscus of water column reaches 100 ml calibration mark. Add 2 ml 20% (v/v) HNO3 and evaporate to dryness in mechanical convection oven (with efficient exhaust to extract acidic vapours). Set effective temperature to 90 ± 5°C.
Prepare several runs and dry in oven. A typical LIS run consists of 38 tubes including 4 blanks, 3 QCs and 2 duplicate spikes (QC solution - 5 ml - added by Brinkmann dispenser). Cool, add 5 ml 5% HNO3 and 1 drop 50% H2O2. Seal tubes with Parafilm, stir with Vortex and hand-turn to wet entire inside surface. Repeat after ½ hour. Transfer supernatant to numbered tube (17 x 100 mm) with cap.
INTERFERENCES: Several (compensated for by computer program).

REPORTING RESULTS:Report > 0.010 ug/ml to 2 sig figs;< 0.010 to 1 sig.
INSTRUMENTATION: Jarrell Ash Atom-Comp ICP Model 975.

Calibration Range: 0.003 to 75 mg/L
Resolution: 0.001 mg/L
Sensitivity:
Instrument Detection Limit: 0.003 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.100 mg/L
Accuracy- 95.8% (QCS, in-house control; X 0.0098 mg/L; N = 35)
Precision of Controls-

	A	B
mean	.205 mg/L	
std. dev.	.014 mg/L	
R.S.D.	6.9 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0008	0.0019	0.0041
mean	0.013	0.032	0.071

W .0005mg/L T .0025mg/L

CONTROL LIMITS:

REMARKS:PE - 5000 AAS available as back-up unit.
For throughfalls, terrestial effect and other APIOS samples, 50 ml volume is concentrated to 5 ml.

SUMMARY REPORT OF QUALITY CONTROL DATA

MANGANESE IN MISCELLANEOUS WATERS

Operating Range = .00020to 0.100 mg/L

IN - RUN DUPLICATES

range	<.00020	.00020to 0.020	0.020to 0.050	0.050to 0.10	> 0.10
no.	2	115	112	46	76
s.w.		0.00080	0.00190	0.00410	
mean		0.01300	0.03280	0.07120	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcs1	1060	0.20490	0.01423	6.94

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	50	.00067	.00093

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Manganese TEST CODE: MNUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BA0
REVISION NO: 85-1 DATE: July, 1985
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml
Container- Plastic bottle with non-metallic cap liner
Preservative- 0.5 ml conc HNO3
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pipette 25 ml sample into a 1 oz flint glass vial with polyethylene lined screw cap. Add 0.5 ml 20% HNO3 to vial. Prepare several sets of 36 vials including 2 blanks, 2 in-house QC and 2 within-run duplicates for LIS runs.
Dry overnight at 85 ± 5°C in forced air convection oven. Cool to room temperature and add 2.5 ml 5% (V/V) HCl to each vial by Oxford dispenser. Shake thoroughly to dissolve soluble salts.
Determine Fe and Mn by AAS using composite standards (0.4, 1.0, & 2 ppm Mn; 2.0, 5.0 & 10.0 ppm Fe).
The computer program allows direct input of results into LIS if P-E 5000 automated AAS system is used.
INTERFERENCES: Ca and Mg can cause severe interference if flame height and air-acetylene ratio are not optimized,
REPORTING RESULTS:2 dec. places if > 1 µg/ml;if < 1 - 3 dec. places
INSTRUMENTATION: Perkin-Elmer P-E 5000 AAS interfaced with PET computer.

Calibration Range: 0 to 2 mg/L (for Mn)
Resolution:
Sensitivity:
Instrument Detection Limit: 0.02 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.200 mg/L
Accuracy- 101% (in-house standard, X = 0.0395 mg/L N = 84)
Precision of Controls-

	A	B
mean	.042 mg/L	
std. dev.	.0024mg/L	
R.S.D.	5.7 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.002	0.020	0.005
mean	0.017	0.067	0.130

W .0005mg/L T .0025mg/L

CONTROL LIMITS:

REMARKS: ICP-AES system may also be used for simultaneous measurement of both Fe and Mn with comparable accuracy and precision.

SUMMARY REPORT OF QUALITY CONTROL DATA

AA-MANGANESE IN MISCELLANEOUS WATERS

Operating Range = .00200to 0.200 mg/L

IN - RUN DUPLICATES

range	<.00200	.00200to 0.040	0.040to 0.100	0.100to 0.20	> 0.2
no.	24	112	62	15	12
s.w.		0.00220	0.02030	0.00450	
mean		0.01740	0.06670	0.12950	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QC	429	0.04190	0.00240	5.73

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	264	.00970	.00520

DATE 86/09/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Mercury TEST CODE:HGUT,HGFT SAMPLE TYPE: Water
UNIT: Biomaterials SUPERVISOR: R. Sadana

METHOD CODE:542BF1,640AF1
REVISION NO: Original DATE: May,1984
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 100 ml minimum (routine 249 ml)
Container-Glass bottle with Teflon cap
Preservative- Nitric acid (< 1%) with pot. dichromate (< 0.5%)
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Place 100 ml sample aliquot in a bacti bottle.
Add 5 ml H_2SO_4, 2.5 ml HNO_3, 2 ml $K_2S_2O_8$ and 1 ml $KMnO_4$ (saturated). Process in batches of 30 or more.
Treat blanks and calibration standards in exactly the same manner as samples.
Place the bottles in a water bath at 85°C for 2 h then cool to room temperature.
The atomic vapour of mercury is generated by the addition of stannous chloride as a reducing agent and its atomic absorption signal measured at 254 nm.

INTERFERENCES: Water vapour, organic solvents

REPORTING RESULTS: 2 significant figures
INSTRUMENTATION: Laboratory Data Control U.V. Monitor (Pharmacia or Milton-Roy)Technicon or Gilson automatic sampler and peristaltic pump.

Calibration Range: 0 to .300 µg/L
Resolution: .005 µg/L (1 division on recorder chart paper)
Sensitivity: .100 µg/L - reads 0.2 absorbance (20 div on chart)
Instrument Detection Limit: 0.003 µg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.003 to 0.300 µg/L
Accuracy- No standards available
Precision of Controls-

	A	B
mean	1.164µg/L	0.952
std. dev.	0.066µg/L	0.103
R.S.D.	5.6 %	10.8 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.005	0.005	0.008
mean	0.037	0.109	0.189

W .01 µg/L T .05 µg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

MERCURY IN MISCELLANEOUS WATERS

Operating Range = .00300to 0.300 ug/L

IN - RUN DUPLICATES

range	<.00300	.00300to 0.060	0.060to 0.150	0.150to 0.30	> 0.3
no.	0	2	45	5	0
s.w.		0.00500	0.00520	0.00810	
mean		0.03700	0.10900	0.18900	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
con g2-3	48	1.16400	0.06570	5.64
con g2-4	259	0.95200	0.10320	10.84

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	153	.00456	.00057

DATE 87/01/05

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Molybdenum TEST CODE: MOUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BE2
REVISION NO: Original DATE: April, 1985
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 1000 ml
Container- One litre plastic bottle with non-metallic cap liner
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pour sample into a 30 x 200 mm quartz test tube held in rack until lower miniscus of water column reaches 100 ml calibration mark. Add 2 ml 20% (v/v) HNO3 and evaporate to dryness in mechanical convection oven (with efficient exhaust to extract acidic vapours). Set effective temperature to 90 ±5°C .
Prepare several runs and dry in oven. A typical LIS run consists of 38 tubes including 4 blanks, 3 QCs, and 2 duplicate spikes (QC solution - 5 ml - added by Brinkmann dispenser). Cool, add 5 ml 5% HNO3 and 1 drop 50% H2O2. Seal tubes with Parafilm, stir with Vortex and hand-turn to wet entire inside surface. Repeat after ½ hour. Transfer supernatent to numbered tube (17 x 100mm) with cap.
INTERFERENCES: Several; compensated for by computer program.

REPORTING RESULTS:2 sig. figs. if > 0.010 µg/ml;if < 0.010 -1 sig fig.
INSTRUMENTATION: Jarell Ash Atom-Comp -ICP Model 975

Calibration Range: 0.005 to 75 mg/L
Resolution: 0.001 mg/L
Sensitivity:
Instrument Detection Limit: 0.005 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.100 mg/L
Accuracy- 94.5% (QCS, in-house control, X = 0.0094 µg/ml, N = 36)
Precision of Controls- A B

mean	.1811mg/L
std. dev.	.0197mg/L
R.S.D.	10.9 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0009	0.0035	0.0034
mean	0.0084	0.0302	0.0905

W .0005mg/L T .0050mg/L

CONTROL LIMITS:

REMARKS: P-E 5000 AAS is available as back-up unit.
In the case of throughfalls, terrestrial effects and other APIOS samples, concentrate 50 ml down to 5 ml.

SUMMARY REPORT OF QUALITY CONTROL DATA

MOLYBDENUM IN MISCELLANEOUS WATERS

Operating Range = .00050to 0.100 mg/L

IN - RUN DUPLICATES

range	<.00050	.00050to 0.020	0.020to 0.050	0.050to 0.10	> 0.1
no.	22	284	3	37	5
s.w.		0.00090	0.00350	0.00340	
mean		0.00820	0.03020	0.09050	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcs1	1062	0.18110	0.01967	10.86

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	176	.00059	.00162

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Nickel TEST CODE: NIUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BE2
REVISION NO: Original DATE: April, 1985
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 1000 ml
Container- One litre plastic bottle with non-metallic cap liner
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pour sample into a 30 x 200 mm quartz test tube held in rack until lower miniscus of water column reaches 100 ml calibration mark. Add 2 ml 20% (v/v) HNO3 and evaporate to dryness in mechanical convection oven (with efficient exhaust to extract acidic vapours). Set effective temperature to 90 ± 5°C.
Prepare several runs and dry in oven. A typical LIS run consists of 38 tubes including 4 blanks, 3 QCs and 2 duplicate spikes (QC solution - 5 ml - added by Brinkmann dispenser). Cool, add 5 ml 5% HNO3 and 1 drop 50% H2O2. Seal tubes with Parafilm, stir with Vortex and hand-turn to wet entire inside surface. Repeat after ½ hour. Transfer supernatant to numbered tube (17 x 100 mm) with cap.
INTERFERENCES: Several; compensated for by computer program.

REPORTING RESULTS:2 sig. figs. if > 0.010 µg/ml;if < 0.010 -1 sig fig.
INSTRUMENTATION: Jarell Ash Atom-Comp -ICP Model 975

Calibration Range: 0.03 to 75 mg/L
Resolution: 0.001 mg/L
Sensitivity:
Instrument Detection Limit: 0.03 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.100 mg/L
Accuracy-96.6% (QCS, in-house control, X = 0.024 mg/L, N = 36)
Precision of Controls-

	A	B
mean	.525 mg/L	
std. dev.	0.045mg/L	
R.S.D.	8.5 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.007	0.003	0.005
mean	0.009	0.026	0.066

W .002 mg/L T .010 mg/L

CONTROL LIMITS:

REMARKS: P-E 5000 AAS is available as back-up unit.
In the case of throughfalls, terrestrial effects and other APIOS samples, concentrate 50 ml down to 5 ml.

SUMMARY REPORT OF QUALITY CONTROL DATA

NICKEL IN MISCELLANEOUS WATERS

Operating Range = .00100to 0.100 mg/L

IN - RUN DUPLICATES

range	<.00100	.00100to 0.020	0.020to 0.050	0.050to 0.10	> 0.1
no.	14	15	249	18	55
s.w.		0.00700	0.00290	0.00500	
mean		0.00860	0.02640	0.06550	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcs1	1055	0.52480	0.04462	8.50

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	39	.01018	.02239

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:SELENIUM TEST CODE:SEUT SAMPLE TYPE: Water
UNIT: Biomaterials SUPERVISOR: R. Sadana

METHOD CODE:510CF3
REVISION NO: DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 100 ml
Container- Glass bottle with bakelite screw cap (16 oz.)
Preservative-1ml conc. HNO3 for sample filling 16 oz. bottle
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-yes % Extracted->90%
Procedure-A twenty ml sample is pipetted into 20x150 mm pyrex test tube. A batch of sixty-eight tubes including samples,blanks, standards and controls are run. These samples are fed to a automated system for measurement of selenium by hydride-FAAS technique.
Samples with selenium concentration exceeding 10 ng/ml are digested by pipetting 20 ml of sample in a 100 ml beaker and adding 4 ml 6:3:1 HNO3:HCLO4:H2SO4. Heat until dense white fumes evolve. Cool, add 0.5 ml of H2O and 2.5 ml of HCl. Transfer the digestate to a test tube 20 calibrated at 20 ml, dilute to mark with DDW, mix well, and analyze.

INTERFERENCES: Excessive concentrations of Cu, Fe, Ni

REPORTING RESULTS:2 dec. if <10, 1 dec. if 10-100, 0 dec. if >100
INSTRUMENTATION: Atomic Absorption Spectrophotometer (Varian 1200) with strip chart recorder, peristaltic pump, auto-sampler, open-ended and heated quartz 'T' cell (0.6 x 10 cm), and gas-liquid separator.
Calibration Range: 0 to 40 ng/ml
Resolution: 0.01 absorbance
Sensitivity: 20 ng/ml gives 0.200 Abs.
Instrument Detection Limit: 1 ng/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.04 mg/L
Accuracy-
Precision of Controls- A B
mean .015 mg/L
std. dev. .0008mg/L
R.S.D. 5.3 %
Precision of Duplicates-low range mid range high range
s.d.
mean
W .001 mg/L T .005 mg/L

CONTROL LIMITS:

REMARKS:-Accuracy= ratio of mean to cert. value of ref. standard X 100

SUMMARY REPORT OF QUALITY CONTROL DATA

SELENIUM IN MISCELLANEOUS WATERS

Operating Range = .00100to 0.040 mg/L

IN - RUN DUPLICATES

range	<.00100	.00100to 0.008	0.008to 0.020	0.020to 0.04	> 0.0
no.	57	0	0	0	0
s.w.		0.00000	0.00000	0.00000	
mean		0.00000	0.00000	0.00000	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
475-5	28	0.01500	0.00080	5.33

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	.00000	.00000

DATE 87/03/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Silver TEST CODE: AGUT SAMPLE TYPE: Water
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:005BF2
REVISION NO: Original DATE: January 10, 1986
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 100 ml
Container- Acid washed plastic
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- The sample is analyzed directly with no pretreatment by graphite furnace atomic absorption spectrophotometry. 20 µL of sample is placed in a graphite furnace fitted with a L'Vov.

INTERFERENCES: Chemical matrix interferences are overcome by choice of analysis program, use of L'Vov platform and addition of KCl.
REPORTING RESULTS: To 4 decimal places, µg/ml
INSTRUMENTATION: Perkin-Elmer 2380 or 603 AAS with HGA 500 or 400 controller and AS-40 autosampler

Calibration Range: 0.0001 to 0.010 mg/L
Resolution: 0.001 absorbance
Sensitivity:
Instrument Detection Limit: 0.0001 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.0100 mg/L
Accuracy-110% at 0.0012 µg/ml (EPA - WP58-1)
Precision of Controls-

	A	B
mean	.0017mg/L	.0048mg/L
std. dev.	.0002mg/L	.0004mg/L
R.S.D.	11.8 %	8.3 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0003	0.0003	0.0004
mean	0.0010	0.0043	0.0059

W .0001mg/L T .0005mg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

SILVER IN MISCELLANEOUS WATERS

Operating Range = .00010to 0.010 mg/L

IN - RUN DUPLICATES

range	<.00010	.00010to 0.002	0.002to 0.005	0.005to 0.01	> 0.0
no.	2	16	7	5	0
s.w.		0.00026	0.00029	0.00043	
mean		0.00100	0.00430	0.00590	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
epal	10	0.00170	0.00020	11.76
epah	11	0.00480	0.00040	8.33

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.

DATE 88/02/02

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Strontium TEST CODE: SRUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BE2
REVISION NO: Original DATE: April, 1985
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 1000 ml
Container- One litre plastic bottle with non-metallic cap liner
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pour sample into a 30 x 200 mm quartz test tube held in rack until lower miniscus of water column reaches 100 ml calibration mark. Add 2 ml 20% (v/v) HNO3 and evaporate to dryness in mechanical convection oven (with efficient exhaust to extract acidic vapours). Set effective temperature to 90 ± 5°C.
Prepare several runs and dry in oven. A typical LIS run consists of 38 tubes including 4 blanks, 3 QCs and 2 duplicate spikes (QC solution - 5 ml - added by Brinkmann dispenser). Cool, add 5 ml 5% HNO3 and 1 drop 50% H2O2. Seal tubes with Parafilm, stir with Vortex and hand-turn to wet entire inside surface. Repeat after ½ hour. Transfer supernatant to numbered tube (17 x 100 mm) with cap.
INTERFERENCES: Several; compensated for by computer program.

REPORTING RESULTS:2 sig. figs. if > 0.010 µg/ml;if < 0.010 -1 sig fig.
INSTRUMENTATION: Jarell Ash Atom-Comp -ICP Model 975

Calibration Range: 0.008 to 75 mg/L
Resolution: 0.001 mg/L
Sensitivity:
Instrument Detection Limit: 0.008 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.100 mg/L
Accuracy-100.0% (QCS, in-house control, X = 0.22 mg/L, N = 109)
Precision of Controls-

	A	B
mean	.265 mg/L	
std. dev.	.0661mg/L	
R.S.D.	25.0 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0030	0.0029	0.0019
mean	0.0119	0.0342	0.0753

W .001 mg/L T .010 mg/L

CONTROL LIMITS:

REMARKS: P-E 5000 AAS is available as back-up unit.
In the case of throughfalls, terrestrial effects and other APIOS samples, concentrate 50 ml down to 5 ml.

SUMMARY REPORT OF QUALITY CONTROL DATA

STRONTIUM IN MISCELLANEOUS WATERS

Operating Range = .00050to 0.100 mg/L

IN - RUN DUPLICATES

range	<.00050	.00050to 0.020	0.020to 0.050	0.050to 0.10	> 0.10
no.	1	25	82	43	200
s.w.		0.00300	0.00290	0.00190	
mean		0.01190	0.03420	0.07530	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcs1	1055	0.26480	0.06614	24.98

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	268	.00214	.00224

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Titanium TEST CODE: TIUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BE2
REVISION NO: Original DATE: April, 1985
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 1000 ml
Container- One litre plastic bottle with non-metallic cap liner
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pour sample into a 30 x 200 mm quartz test tube held in rack until lower miniscus of water column reaches 100 ml calibration mark. Add 2 ml 20% (v/v) HNO3 and evaporate to dryness in mechanical convection oven (with efficient exhaust to extract acidic vapours). Set effective temperature to 90 ± 5°C.
Prepare several runs and dry in oven. A typical LIS run consists of 38 tubes including 4 blanks, 3 QCs and 2 duplicate spikes (QC solution - 5 ml - added by Brinkmann dispenser). Cool, add 5 ml 5% HNO3 and 1 drop 50% H2O2. Seal tubes with Parafilm, stir with Vortex and hand-turn to wet entire inside surface. Repeat after ½ hour. Transfer supernatant to numbered tube (17 x 100 mm) with cap.
INTERFERENCES: Several; compensated for by computer program.

REPORTING RESULTS:2 sig. figs. if > 0.010 µg/ml;if < 0.010 -1 sig fig.
INSTRUMENTATION: Jarell Ash Atom-Comp -ICP Model 975

Calibration Range: 0.05 to 75 mg/L
Resolution: 0.001 mg/L
Sensitivity:
Instrument Detection Limit: 0.05 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.100 mg/L
Accuracy-100.0% (QCS 1, in-house control, X = 0.20 mg/L, N = 109)
Precision of Controls-

	A	B
mean	.186 mg/L	
std. dev.	.0189mg/L	
R.S.D.	10.2 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0007	0.0081	0.0052
mean	0.0096	0.026	0.090

W .002 mg/L T .020 mg/L

CONTROL LIMITS:

REMARKS: P-E 5000 AAS is available as back-up unit.
In the case of throughfalls, terrestrial effects and other APIOS samples, concentrate 50 ml down to 5 ml.

SUMMARY REPORT OF QUALITY CONTROL DATA

TITANIUM IN MISCELLANEOUS WATERS

Operating Range = .00100to 0.100 mg/L

IN - RUN DUPLICATES

range	<.00100	.00100to 0.020	0.020to 0.050	0.050to 0.10	> 0.10
no.	27	262	16	31	15
s.w.		0.00070	0.00810	0.00520	
mean		0.00960	0.02630	0.08970	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcs1	1037	0.18610	0.01890	10.16

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	.00000	.00000

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Uranium TEST CODE: UUUT SAMPLE TYPE: Water
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:001BE5
REVISION NO: 1 DATE: January 24, 1986
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml
Container- Polyethylene container
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted->100
Procedure- Samples are analyzed without any pretreatment. Samples are poured from sample containers into 15 ml polystyrene test tubes and analyzed by the ICP-MS technique.

INTERFERENCES: High dissolved solids as found in some industrial waste matrices.
REPORTING RESULTS: µg/l
INSTRUMENTATION: Elan 250 (ICP/MS)

Calibration Range: 0 to 1.000 mg/L
Resolution: 1.0 a.m.u.
Sensitivity: 0.100 mg/L std. produces 30,000-85,000 counts/sec.
Instrument Detection Limit:.0001 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.010 mg/L
Accuracy- Within 10% of NBS
Precision of Controls-

	A	B
mean	.0105mg/L	
std. dev.	.0005mg/L	
R.S.D.	4.37 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.00007	0.00005	
mean	0.00038	0.00802	

W .0001mg/L T .0005mg/L

CONTROL LIMITS:

REMARKS: Replaces fluorometric method

SUMMARY REPORT OF QUALITY CONTROL DATA

URANIUM IN MISCELLANEOUS WATERS

Operating Range = .00001to 0.010 mg/L

IN - RUN DUPLICATES

range	<.00001	.00001to 0.002	0.002to 0.005	0.005to 0.01	> 0.01
no.	0	16	0	1	1
s.w.		0.00007	0.00000	0.00005	
mean		0.00038	0.00000	0.00802	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
comp	74	0.01051	0.00047	4.37

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	62	.00003	.00003

DATE 87/01/14

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Vanadium TEST CODE: VVUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BE2
REVISION NO: Original DATE: April, 1985
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 1000 ml
Container- One litre plastic bottle with non-metallic cap liner
Preservative- 1 ml conc HNO_3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pour sample into a 30 x 200 mm quartz test tube held in rack until lower miniscus of water column reaches 100 ml calibration mark. Add 2 ml 20% (v/v) HNO_3 and evaporate to dryness in mechanical convection oven (with efficient exhaust to extract acidic vapours). Set effective temperature to 90 ± 5°C.
Prepare several runs and dry in oven. A typical LIS run consists of 38 tubes including 4 blanks, 3 QCs and 2 duplicate spikes (QC solution - 5 ml - added by Brinkmann dispenser). Cool, add 5 ml 5% HNO_3 and 1 drop 50% H_2O_2. Seal tubes with Parafilm, stir with Vortex and hand-turn to wet entire inside surface. Repeat after ½ hour. Transfer supernatant to numbered tube (17 x 100 mm) with cap.
INTERFERENCES: Several; compensated for by computer program.

REPORTING RESULTS:2 sig. figs. if > 0.010 µg/ml;if < 0.010 -1 sig fig.
INSTRUMENTATION: Jarell Ash Atom-Comp -ICP Model 975

Calibration Range: 0.008 to 75 mg/L
Resolution: 0.001 mg/L
Sensitivity:
Instrument Detection Limit: 0.008 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.100 mg/L
Accuracy- 95.7% (QCS, in-house control, X = 0.19 mg/L, N = 36)
Precision of Controls-

	A	B
mean	.194 mg/L	
std. dev.	.0146mg/L	
R.S.D.	7.5 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0008	0.0006	0.0032
mean	0.0096	0.0207	0.0926

W .0005mg/L T .0050mg/L

CONTROL LIMITS:

REMARKS: P-E 5000 AAS is available as back-up unit.
In the case of throughfalls, terrestrial effects and other APIOS samples, concentrate 50 ml down to 5 ml.

SUMMARY REPORT OF QUALITY CONTROL DATA

VANADIUM IN MISCELLANEOUS WATERS

Operating Range = .00050to 0.100 mg/L

IN - RUN DUPLICATES

range	<.00050	.00050to 0.020	0.020to 0.050	0.050to 0.10	> 0.10
no.	14	288	5	31	13
s.w.		0.00080	0.00060	0.00320	
mean		0.00940	0.02070	0.09260	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcs1	1056	0.19440	0.01460	7.51

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	18	.00075	.00117

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Zinc TEST CODE: ZNUT SAMPLE TYPE: Waters
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:522BE2
REVISION NO: Original DATE: April, 1985
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 1000 ml
Container- One litre plastic bottle with non-metallic cap liner
Preservative- 1 ml conc HNO3 per litre.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pour sample into a 30 x 200 mm quartz test held in rack until lower minicus of water column reaches 100 ml calibration mark. Add 2 ml 20% (v/v) HNO3 and evaporate to dryness in mechanical convection oven (with efficient exhaust to extract acidic vapours). Set effective temperature to 90 ±5°C.
Prepare several runs and dry in oven. A typical LIS run consists of 38 tube including 4 blanks, 3 QCs and 2 duplicate spikes (QC solution - 5 ml - added by Brinkmann dispenser). Cool, add 5 ml 5% HNO3 and 1 drop 50% H2O2. Seal tubes with Parafilm, stir with Vortex and hand-turn to wet entire inside surface. Repeat after ½ hour. transfer supernatent to numbered tube (17 x 100 mm) with cap.
INTERFERENCES: Several; compensated for by computer program.

REPORTING RESULTS:2 sig. figs. if > 0.010 µg/ml;if < 0.010 -1 sig fig.
INSTRUMENTATION: Jarell Ash Atom-Comp -ICP Model 975

Calibration Range: 0.005 to 75 mg/L
Resolution: 0.001 mg/L
Sensitivity:
Instrument Detection Limit: 0.005 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.100 mg/L
Accuracy- 92.7% (QCS, in-house control, X = 0.0093 µg/ml, N = 35)
Precision of Controls-

	A	B
mean	.2189mg/L	
std. dev.	.0342mg/L	
R.S.D.	15.6 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0018	0.0050	0.0246
mean	0.0135	0.0305	0.0707

W .0005mg/L T .0025mg/L

CONTROL LIMITS:

REMARKS: P-E 5000 AAS is available as back-up unit.
In the case of throughfalls, terrestrial effects and other APIOS samples, concentrate 50 ml down to 5 ml.

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC IN MISCELLANEOUS WATERS

Operating Range = .00050to 0.100 mg/L

IN - RUN DUPLICATES

range	<.00050	.00050to 0.020	0.020to 0.050	0.050to 0.10	> 0.10
no.	9	165	93	19	65
s.w.		0.00180	0.00500	0.02460	
mean		0.01350	0.03050	0.07070	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcs1	1017	0.21890	0.03418	15.61

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	367	.00351	.01372

DATE 87/03/20

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Total Cyanide TEST CODE: CCNAUR SAMPLE TYPE: Water
UNIT: QC-Project SUPERVISOR: J. Hipfner

METHOD CODE:001AC2
REVISION NO: DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml
Container- Glass or plastic (preferred)
Preservative- NaOH
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.- % Extracted-100
Procedure- The sample is screened using the automated high temp. distillation with 25% H3PO4-5% H3PO2 followed by a colourimetric analysis with chloramine T -isonicotinic acid -barbituric acid method.

If the total cyanide is > .01 mg/L then 5 to 250 ml of sample is manually distilled with 30 ml of 15%(w/v)tartaric acid. The distillate is collected in 50 ml of 1N NaOH, and analyzed by the automated Technicon distillation system referred to above.

INTERFERENCES: SCN interference is removed by distillation. Distillable organics may interfere; also S= at high levels.
REPORTING RESULTS:Mg/l CN: 3 decimal places up to 3 significant figs
INSTRUMENTATION: Technicon AAII continuous flow analyzer including pump, colourimeter, appropriate autosampler and recorder. High temperature distillation apparatus (Technicon). Manual dist. app
Calibration Range: 0 to 0.4 mg/l as CN
Resolution: 0.001 mg/l
Sensitivity:
Instrument Detection Limit: 0.001 mg/l
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.400 mg/l
Accuracy- 100%
Precision of Controls-

	A	B
mean	.110 mg/L	0.059
std. dev.	.0027mg/L	0.0026
R.S.D.	2.45 %	4.41 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0017	0.0063	0.0074
mean	0.025	0.134	0.281

W .001mg/L T .005mg/L

CONTROL LIMITS:

REMARKS: Pure CN standards are recovered 100% during manual distillation. Complex cyanides can normally be expected to be recovered at 100%.

SUMMARY REPORT OF QUALITY CONTROL DATA

TOTAL CYANIDE IN MISCELLANEOUS WATERS

Operating Range = .00100to 0.400 mg/L

IN - RUN DUPLICATES

range	<.00100	.00100to 0.080	0.080to 0.200	0.200to 0.40	> 0.40
no.	0	56	5	1	15
s.w.		0.00080	0.00450	0.02830	
mean		0.01170	0.15670	0.26000	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qc-a	146	0.14900	0.00490	3.29
qc-b	146	0.01800	0.00220	12.22

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	146	.00100	.00000

DATE 88/06/02

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Free cyanide TEST CODE: CCNFUR SAMPLE TYPE: Water
UNIT: QC-Project SUPERVISOR: J. Hipfner

METHOD CODE:700AC2
REVISION NO: DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 100 ml
Container- Glass or plastic (preferred)
Preservative- NaOH
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.- % Extracted- *
Procedure- Pass sample aliquot through an automated low temperature distillation (106°C) in a distillation acid consisting of 10% acetic acid and 0.5% zinc acetate.
Analyze distillate by the Chloramine-T -pyridine-barbituric acid colourometric method, or equivalent.

INTERFERENCES: None

REPORTING RESULTS:Mg/l CN to 3 decimal places up to 3 significant figs
INSTRUMENTATION: Technicon automated continuous flow analyzer including pump, colourimetric distillation apparatus and sampler; suitable recorder.
Calibration Range: 0 to 0.4 mg/l as CN
Resolution: 0.001
Sensitivity:
Instrument Detection Limit: 0.001 mg/l
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0 to 0.400 mg/L
Accuracy- 100%
Precision of Controls-

	A	B
mean	.110 mg/L	0.060
std. dev.	.0036mg/L	0.0031
R.S.D.	3.27 %	5.17%

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0026	0.0021	0.0028
mean	0.022	0.161	0.209

W .001 mg/L T .005 mg/L

CONTROL LIMITS:

REMARKS:* The test defines the results reported in this case. The terminology "Weak Acid Dissociable" is commonly used and represents weakly associated cyanide compounds such as KCN, NaCN, NiCN4, HCN,etc.

SUMMARY REPORT OF QUALITY CONTROL DATA

FREE CYANIDE IN MISCELLANEOUS WATERS

Operating Range = .00100to 0.400 mg/L

IN - RUN DUPLICATES

range	<.00100	.00100to 0.080	0.080to 0.200	0.200to 0.40	> 0.4
no.	1	53	4	0	14
s.w.		0.00090	0.00320	0.00000	
mean		0.01080	0.15560	0.00000	

QA Control Samples

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qc-a	135	0.15100	0.00620	4.11
qc-b	135	0.01800	0.00220	12.22

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	135	.00100	.00000